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WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 3 :
B05D 1/34, 1/36, 3/04
B05B 3/10, 5/02, 7/06
B05B 12/08 // B05D 7/00, 7/06
B05D 7/14

A1

(11) International Publication Number: WO 84/ 02665

(43) International Publication Date: 19 July 1984 (19.07.84)

(21) International Application Number: PCT/AU83/00196

(22) International Filing Date: 30 December 1983 (30.12.83)

(31) Priority Application Number: PF 7466

(32) Priority Date: 31 December 1982 (31.12.82)

(33) Priority Country: AU

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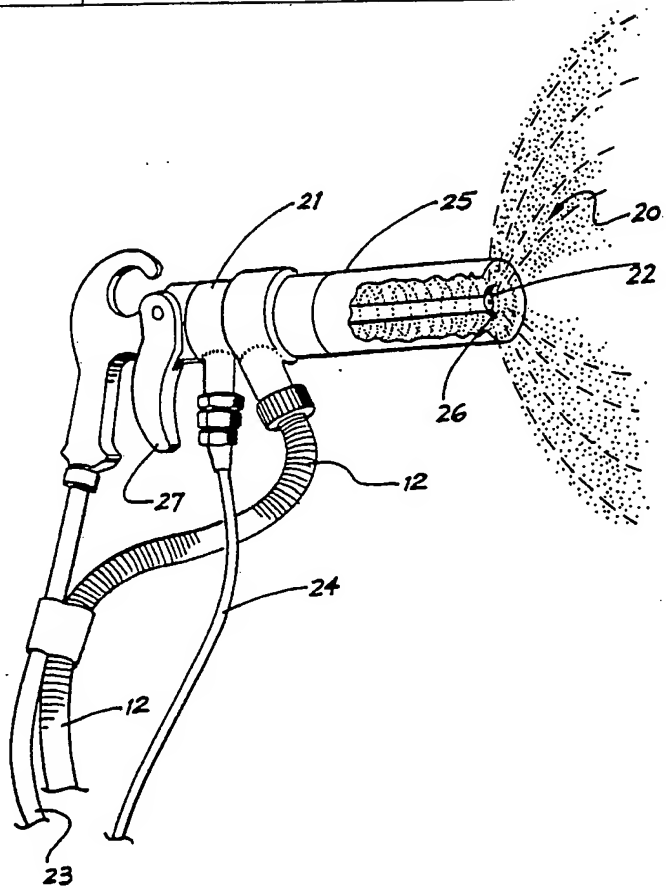
(81) Designated States: AT, BR, CH, DE, DK, FI, GB, HU, JP, LU, NL, NO, RO, SE, SU, US.

Published
With international search report.

(54) Title: COATING PROCESS

(57) Abstract

A process of drying or curing coatings and films of paints, lacquers, varnishes, printing vehicles and inks. A substrate is coated in any appropriate manner by a vehicle such as a one-or two-component paint and exposed to a drying or curing agent which is deposited electrostatically on the coated vehicle. The steps of coating the substrate with the vehicle and deposition of the drying agent are performed either simultaneously or sequentially. Apparatus includes an electrostatic spray gun (21) with a barrel (22) and a concentrically located shroud (25) ensuring the atomised drying agent is not emitted before the tip (26) of the barrel is reached.



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COATING PROCESS

The invention relates to the drying of coatings, films and the like. By the invention there is provided an improved process (and resultant product) whereby said drying is carried out more efficaciously then heretofore.

In one broad aspect, the invention provides a process for forming a dried coating upon a suitable substrate comprising coating a vehicle upon said substrate, and subjecting the coated vehicle to treatment with a drying agent, the said agent being electrostatically deposited upon the coated vehicle. The invention has other aspects as will be evidenced hereafter.

The invention finds application in the drying of paints, lacquers, varnishes, printing vehicles and printing inks, liquid adhesives, surface coatings, caulking compounds and the like. In the above definition, the following are to be understood:

1. In respect of coating, film or the like - which is to be, or has been, subjected to the process of the invention - the term "drying" is to be understood as (i) including within its ambit "curing" and as (ii) indicating that the coating is either free from "tack", insoluble in solvent, possessed of an advanced degree of integrity, or able to withstand reasonable abrasion or pressure without damage. It will also be appreciated that, in some circumstances, a dry coating may evidence all of the foregoing qualities. The expression "coating", when used as a noun, is, for the purposes of this invention, to be understood as synonymous with "film" (or the like).
2. The expression "substrate" is to be construed in the widest possible sense, any surface to which the

vehicle can be adheringly applied, and upon which it will be retained while treatment with the agent is being effected, being within the ambit of the invention. Thus, such wide-ranging materials as paperboard, metal foil, steel plate, plastic material, thermally sensitive material and so on may (depending upon other circumstances) be employed.

3. The expression "vehicle" includes within its ambit the paints et al as particularised heretofore.

4. The expression "agent" connotes the at least one chemical compound which effect(s) the curing or drying of the vehicle. It may be sometimes alternatively referred to, in this text, as a catalytic agent, or simply as a catalyst.

In one form of the invention, the vehicle may be of the type which contains free isocyanate groups. The expression "free isocyanate groups" includes within its ambit potentially free such groups, the meaning to be conveyed being that the pre-polymer has isocyanate groups which are releasable, or available, for reaction with any other compound possessing active hydrogen sites (for the purposes of polymer propagation and/or film formation). Compounds containing free isocyanate groups are to be understood as embracing all such compounds. Accordingly, comprehended thereby are, not only isocyanates with urethane structure and polyisocyanates, but also those with polyisocyanurate, biuret, and allophanate structure.

The drying (or catalytic) agent, which may effect its treatment in vapour-phase, may be ammonia, or an amine, or any other compound, such as organo metals, or inorganic metal salts, capable of accelerating the desired reaction pathway. The

expression "amine" includes within its ambit not only those of simple primary aliphatic monofunctional structure, but also amines characterised by (i) polyfunctionality and (ii) a more advanced degree of hydrogen substitution. The expression "vapour-phase" denotes that the agent - namely ammonia, an amine et al - is in gaseous, vapour, or any other entrained air-borne form (e.g. dispersion, fog or aerosol) in which it is available for reaction.

The amine itself may be widely examplified. Thus, typical examples are mono compounds such as methylamine, ethylamine, propylamine, isopropylamine and the numerous isomers of butylamine, and polyfunctional amines such as hydrazine, ethylenediamine, propylene diamine and diethylene triamine. Further examples are diethylamine, triethylamine and dimethylethanolamine (DMEA), and ditertiary amines such as N,N,N',N'-tetramethylethylenediamine (TMEDA) and N,N,N',N',2-pentamethyl-1,2-propanediamine (PMT) - and, indeed, any combination of such amines, proportioned as required, whereby advantage may be taken of the synergistic effect of such a combination.

The organo metals may also be widely exemplified. Thus, typical examples are dibutyl tin dilaurate, lead tetraethyl, titanium acetyl acetonate, dimethyl tin dichloride, and stannous and zinc octoates. Among the inorganic metal salts shown to be effective, there may be mentioned bismuth nitrate and ferric chloride. Likewise, advantage can be taken of the synergistic effect of these compounds in association both with one another and with the above-mentioned amine(s).

The vehicle may be a one- or two- component paint et al which contains free isocyanate groups (as defined above). A typical such paint - which is able to be electrostatically or

otherwise deposited upon a substrate to be coated, and speedily dried by a vapour-phase drying agent as further demonstrated hereafter - is a two-component preparation formulated from a hydroxyl-bearing synthetic resinous first component, and an isocyanate terminated pre-polymer second component. It will be appreciated that these components are themselves capable of wide exemplification.

One suitable such paint is a two-component white polyurethane preparation in which pigment dispersion has been carried out using a coconut alkyd based resin which is subsequently mixed with an isocyanate terminated pre-polymer based on XDI (xylene diisocyanate). In alternative formulations, the XDI-based isocyanate terminated pre-polymer can be replaced by one or more pre-polymer(s) based on (using the standard abbreviations) MDI, TDI, HDI, H_{12}MDI, IPDI, and H_6XDI - or the reaction products of these diisocyanate monomers with appropriate polyols, polycarboxy or polyamine intermediates. Likewise, the first component can be alternatively chosen from inter alia (using generic designations) acrylic, epoxy, polyether, polyester and polysiloxane resins.

A further exemplary vehicle, which is able to be electrostatically or otherwise coated upon the substrate, and speedily dried in accordance with the invention, is a two-part vehicle in which the first part comprises a polyepoxide resin containing hydroxyl groups, and the second part comprises a resin containing free polyamide groups.

The electrostatic deposition of the drying or catalytic agent may be carried out by subjecting the vapourized agent to a generated electrostatic field. In one form of the invention, the electrostatic field may be established in

orthodox fashion, typically per medium of an electrostatic gun of the type conventionally known for that purpose.

In one embodiment of the invention, electrostatic deposition of the drying agent (catalyst) may follow a preceding step of coating the vehicle upon the substrate. However co-deposition, including electrostatic co-deposition - that is, simultaneous application of both coating and drying agent by action of an electrostatic field - is also within the ambit of the invention. Thus, in a further process aspect, the invention provides a process as broadly defined heretofore, and further characterised in that the steps of coating the vehicle upon the substrate, and electrostatically depositing the drying agent, are carried out simultaneously. In a related aspect, the invention provides apparatus, as set forth hereafter, for carrying out this process.

As a prelude to defining the just-mentioned apparatus, reference is made to the manner in which the vehicle is coated upon the substrate. In this connection the vehicle, typically a paint, may be so coated by hand painting (using a brush), dipping, spraying - or by using apparatus whereby the coating of the paint upon the substrate is itself electrostatically performed. Such electrostatic deposition of paint may be effected by means of an electrostatic paint gun.

The apparatus for carrying out the co-deposition of vehicle and drying agent may do so (as indicated above) by action of an electrostatic field. In this related aspect of the invention, the invention provides apparatus which comprises, in combination, means for directing electrostatically charged vehicle, such as a paint, upon the substrate to be coated; and means for simultaneously directing an electrostatically charged

drying agent, typically in vapour-phase, upon the substrate, the first and second-mentioned means being concentrically arranged in relation to one another. This apparatus, which can also function to direct drying agent only upon the substrate, will be described in greater detail hereafter.

The vapour-phase drying agent can be generated in a variety of ways, including evaporative or injection techniques. Apparatus whereby drying agent in vapour-phase is efficiently produced forms another aspect of the invention. In this aspect, the invention provides apparatus which comprises, in combination, means for vapourizing a liquid drying agent; means for controllably delivering the vapourized agent to a required location - for example to the second-mentioned means of the apparatus as defined above; and sensing means provided in the delivery path and operative to ensure that the concentration of delivered vapourized agent is maintained within pre-determined limits. When the vapourized agent is delivered to the said second-mentioned means, the vapour-phase generating components and the co-deposition components combine to function as a single apparatus.

The invention will now be sequentially described with reference to the accompanying drawings, and some specific numerical examples. It will be understood that such ensuing description is intended to illustrate performance features of the invention - and hence is not to be limitatively construed.

In the drawings:

Figure 1 is a perspective-type view of the apparatus for providing (generating) the drying agent in vapour-phase.

Figure 2 is a perspective-type view of the apparatus for effecting deposition of drying agent - or co-deposition of

vehicle and drying agent.

The apparatus of Figure 1 (generally designated by the numeral 1) is comprised of a box-like outer structure 2 containing a tank 3 for the liquid catalyst. The catalyst is atomized within an inner chamber 4, situated beneath tank 3, by means of atomizing nozzles 5 which receive catalyst from the tank 3 under gravity. Air is admitted to the chamber 4 via an air inlet filter 7 in a side 8 of the structure. Within the chamber 4, a turbulent air flow is created in order to facilitate mixing and atomization therein. This is achieved by a turbulence-creating fan 6 in the base of the chamber.

The atomizing nozzles 5, whilst being fed with catalyst from the tank 3, receive compressed air via a hose 9 which is used to produce a fine atomizing spray at the nozzle 5.

To deliver the vapour-phase catalyst from the chamber 4, a variable-speed turbulence fan 10, the operation of which is controlled from mechanism 16, is located in a side 11 of the structure opposite the side 8 containing the air filter 7. The fan 10 directs vapour-phase catalyst to a required location - in a particular instance to the co-deposition apparatus as defined above and described hereafter with reference to Figure 2 - via a flexible conduit 12. Located in a cowling 13 disposed around the fan 10 is a catalyst sensor 14. This sensor measures the concentration of vapour-phase catalyst passing along the conduit and provides a concentration reading which is fed back to, and recorded on, a dial 15.

By adjusting the atomization and vapourization of the liquid catalyst stored in the tank 3, which can be controlled within pre-determined limits from the dial 15, the concentration of the vapour-phase catalyst being delivered from the

apparatus can be monitored as required. Also, as indicated heretofore, the rate of delivery of the catalyst is controlled by operation of the variable speed fan 10. In this manner, preset concentrations of catalyst can be maintained with accuracy.

In the case of molecular solution of the vapour-phase catalyst in air, the concentrations will of course vary between zero and the saturation concentration for the particular catalyst used at the temperature under consideration. In the case of aerosol fogs this restriction does not exist.

As indicated above, the apparatus of Figure 1 can use flexible conduit 12 to feed vapour-phase catalyst to the apparatus illustrated in Figure 2 (and generally designated by the numeral 20). This apparatus can be used for electrostatic deposition of catalyst only, or for the electrostatic co-deposition of catalyst and a vehicle - typically paint.

The apparatus 20 comprises a standard electrostatic gun 21 having a barrel 22 from whence the electrostatic charge is emitted. Feeding into the rear of the gun 21 is a supply conduit 23 which delivers the paint to the barrel 22 of the gun, from which it is likewise emitted with an electrostatic charge. Power lead 24 provides the power for the production of the electrostatic charge in the gun 21. Thus far, the gun is conventional - and well known in the art.

As indicated above, the flexible conduit 12 can connect the apparatus of Figure 1 to the gun 21. The vapour-phase catalyst is projected to the barrel of the electrostatic gun by means of the pressure difference created by the fan 10 of Figure 1. In the apparatus of Figure 2, a barrel shroud 25, concentrically deposed around the barrel 22, ensures that the

vapour-phase catalyst is not emitted from the gun 21 before the tip 26 of the barrel is reached. In this way, the catalyst is also charged by the electrostatic field produced at the tip of the barrel 22 - and is charged sufficiently to allow the vapourized, and now charged, catalyst to deposit itself on an earthed substrate at which the gun is being pointed.

If the gun is to be used, in one mode, for electrostatic deposition of drying agent only (following previous painting of the substrate), actuation of trigger mechanism 27 will achieve this end. If, in another mode, co-deposition is required, the gun 20, controlled by operation of trigger mechanism 27,will be simultaneously supplied with paint via conduit 23, vapour-phase catalyst via conduit 12 (and electrostatic charge via lead 24). When the trigger 27 is depressed in this mode, paint from the barrel 20 and catalyst from the shroud 25 will be simultaneously electrostatically charged. Both the flow of paint and the flow and concentration of catalyst can be controlled to achieve a desired ratio in the co-deposition of paint and catalyst. It will of course be readily appreciated that, when the components of Figure 1 deliver the drying agent in vapour-phase to the components of Figure 2, via flexible conduit 12, the so associated components function as a single apparatus.

The process by which electrostatically charged catalyst is applied to pre-painted articles (to all surfaces thereof) or where co-deposition of electrostatically charged catalyst and paint is effected (to all such surfaces), results in a significant acceleration of the curing of the paint film, to give drying times of obvious commercial significance. The manner in which deposition or co-deposition is effected at all surfaces will be understood from Figure 2 - which depicts the

divergent path of drying agent and vehicle as the said agent and vehicle leave the apparatus.

The invention will now be described with reference to five numerical examples. In relation thereto, the following should be noted.

The paint employed in these examples is a two-component white polyurethane preparation as mentioned hereinbefore.

In so far as it is of known structure, the electrostatic gun is of three basic types - which, for convenience, are referred to hereafter as types I, II and III respectively. Briefly, the type I gun performs the electrostatic deposition of paint (or other vehicle) by means of a rotating disc which atomizes the paint within an electrostatic field generated at the tip of a wire filament disposed for that purpose (the thus charged paint being delivered to the substrate being coated). The type II gun generates an electrostatic field per medium of a filament disposed at the end of a barrel, through which is delivered the paint to be carried by the electrostatic charge (the paint being delivered to and through the barrel by means of air assistance). The type III gun operates in substantially the same manner as the type II gun; however, the paint to be electrostatically charged is delivered to the barrel hydraulically.

EXAMPLE 1 (sequential deposition)

A metal panel, earthed properly, is coated on both faces with a paint as previously described, using an electrostatic hand type I gun. Air containing approximately 2,000 parts per million of dimethylethanolamine in vapour form which has been generated from the apparatus of Figure 1, is passed at right angles to the painted plate - and within two minutes of elapsed

time, the vapour-phase catalyst is charged using an electrostatic type III gun, with no paint being supplied to the gun, and only a charge being generated. The gun is arranged opposite the plate thus allowing the charged field to intersect the catalyst flow. The passage of vapour-phase catalyst and the electrostatic field from the type III gun is run continuously for approximately two minutes after which time both catalyst and electrostatic charge are discontinued. After a further eight minutes post-curing time in slightly turbulent air, the film is found to have been accelerated in drying on both sides of the plate to give a satisfactorily dried film.

EXAMPLE 2 (sequential deposition)

Vapourized dimethylethanolamine (DMEA hereafter) from the apparatus of Figure 1 is passed through flexible tube 12 to plastic barrel shroud 25 around the barrel 22 of an electrostatic gun. This is the apparatus of Figure 2 with the gun, in this case, being an electrostatic type II gun. Painted panels prepared in a manner as described in Example 1 above, are exposed to a flow of the vapour-phase catalyst from the gun as illustrated in Figure 2. The vapour-phase catalyst is applied at a concentration of approximately 2,500 parts per million, for approximately sixty seconds after painting. This flow of catalyst is maintained, for approximately two minutes, after which it is cut out and the plate exposed to gently turbulent air for approximately eight minutes. After this post-curing period, drying of the painted film is found to have been markedly accelerated on both sides of the plate.

EXAMPLE 3 (co-deposition)

A deposition gun, as described in Example 2 and illustrated in Figure 2, is set up and connected to the apparatus of Figure

1. Paint, as previously described, is then passed through the gun - and simultaneously, the vapour-phase catalyst (DMEA) was introduced in a concentration of approximately 4,000 parts per million of DMEA as a molecular solution in air. Properly earthed panels were painted by the simultaneous application of paint and catalyst. Under these conditions, the painted plates showed equally rapid attainment of touch-dry condition and overall commercial dryness.

EXAMPLE 4

This example is similar to that described in Example 3 above, i.e. the same procedures were repeated but in this instance, the vapour-phase catalyst employed was PMT (all other parameters were the same). The degree of accelerated drying was even more marked than with DMEA and an even more rapid commercial dryness was effected.

EXAMPLE 5

In this example, the vapour-phase catalyst was lead tetraethyl. All other conditions as detailed in Example 3 above were idential. Once again an equally marked acceleration in the curing of the paint was achieved.

In closing, it is reiterated that the foregoing detailed description is intended to be merely illustrative of the invention. As long as the basic criteria are observed, matters falling there within, not being critical in themselves, can be varied in accordance with situational requirements.

THE CLAIMS:

1. A process for forming a dried coating upon a suitable substrate comprising coating a vehicle upon said substrate, and subjecting the coated vehicle to treatment with a drying agent, said agent being electrostatically deposited upon the said coated vehicle.

2. A process as claimed in claim 1, wherein the steps of coating the vehicle upon the substrate, and electrostatically depositing the drying agent, are carried out either sequentially or simultaneously.

3. A process as claimed in claim 1 or 2, wherein the drying agent carries out its drying treatment in vapour-phase.

4. A process as claimed in any one of claims 1 to 3, in which the vehicle is a one-or two-part vehicle which contains free isocyanate groups.

5. A process as claimed in claim 3 or 4, wherein the drying agent is selected from ammonia, an amine, an organo metal catalyst, or any combination thereof.

6. A process as claimed in claim 5, wherein the drying agent is an alkanolamine.

7. A process as claimed in claim 3, or any one of claims 4 to 6 when appended thereto, in which the drying agent is prepared in vapour-phase by controlled atomization of a precursor liquid drying agent.

8. A process for forming a dried coating upon a suitable substrate comprising the steps of coating a vehicle upon the substrate, and subjecting the vehicle to treatment with a drying agent in vapour-phase; wherein the said drying agent is electrostatically deposited upon the vehicle and

(i) the step of coating the vehicle upon the substrate precedes the step of electrostatically depositing the drying agent in vapour-phase; or

(ii) the step of coating the vehicle upon the substrate and the step of electrostatically depositing the drying agent in vapour-phase are carried out simultaneously.

9. A process as claimed in claim 8 in which the vehicle is a one- or two-part vehicle which contains free isocyanate groups.

10. A process as claimed in claim 8 or 9 wherein the vehicle is electrostatically coated upon the substrate.

11. A process as claimed in claim 1 or 8 substantially as herein described with reference to any one of the foregoing examples and/or accompanying drawings.

12. Apparatus for use in carrying out the process of claim 1 or 8 comprising, in combination, means for directing electrostatically charged vehicle, such as a paint, upon the substrate to be coated; and means for simultaneously directing an electrostatically charged drying agent in vapour-phase upon the substrate, the first and second-mentioned means being concentrically arranged in relation to one another.

13. Apparatus for use in carrying out the process of claim 1 or 8 comprising, in combination, means for vapourizing a liquid drying agent; means for controllably delivering the vapourized agent to a required location; and sensing means provided in the delivery path and operative to ensure that the concentration of delivered vapourized agent is maintained within pre-determined limits.

14. Apparatus as claimed in claim 13 wherein the required location is the apparatus of claim 12, the vapourized agent being controllably delivered to the second-mentioned means of the last-mentioned apparatus.

15. Apparatus as claimed in any one of claims 12 to 14, substantially as herein described with reference to Figures 1 and 2 of the accompanying drawings.

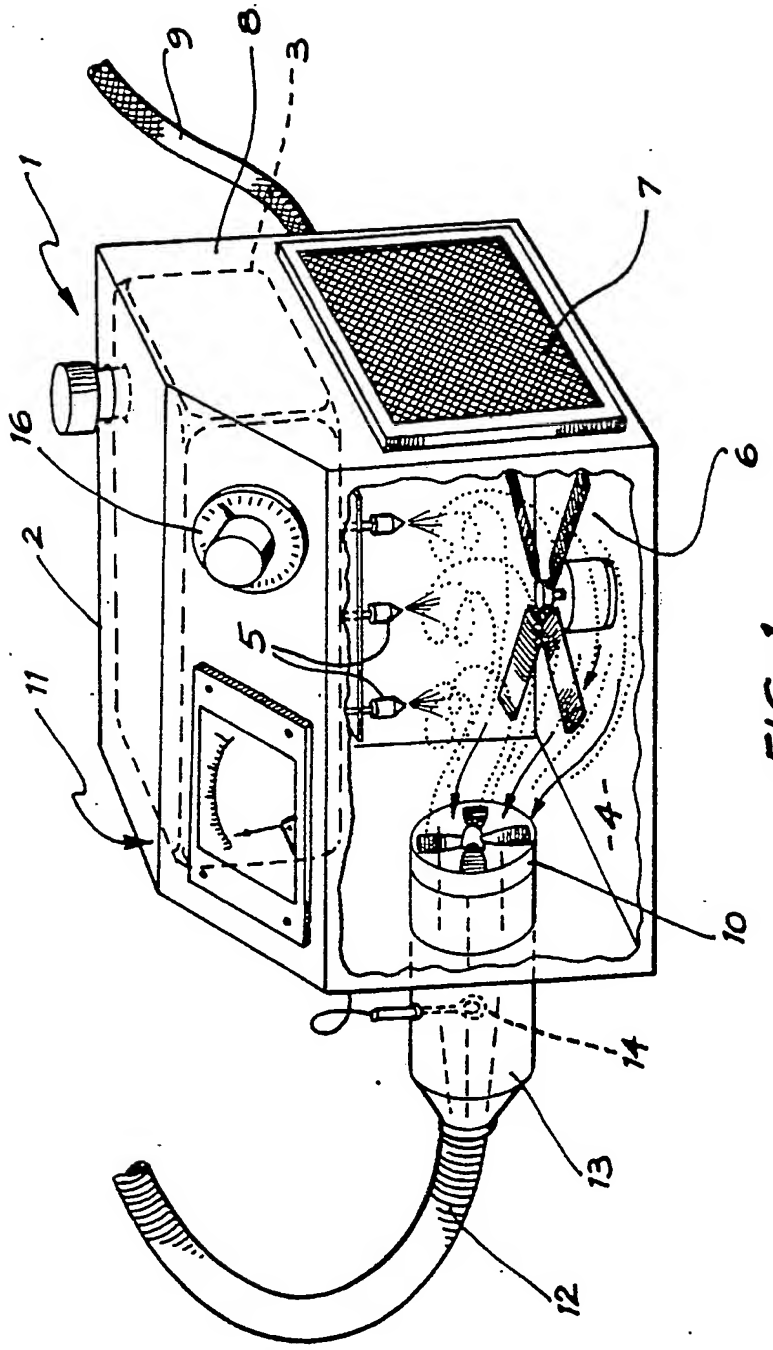

FIG. 1

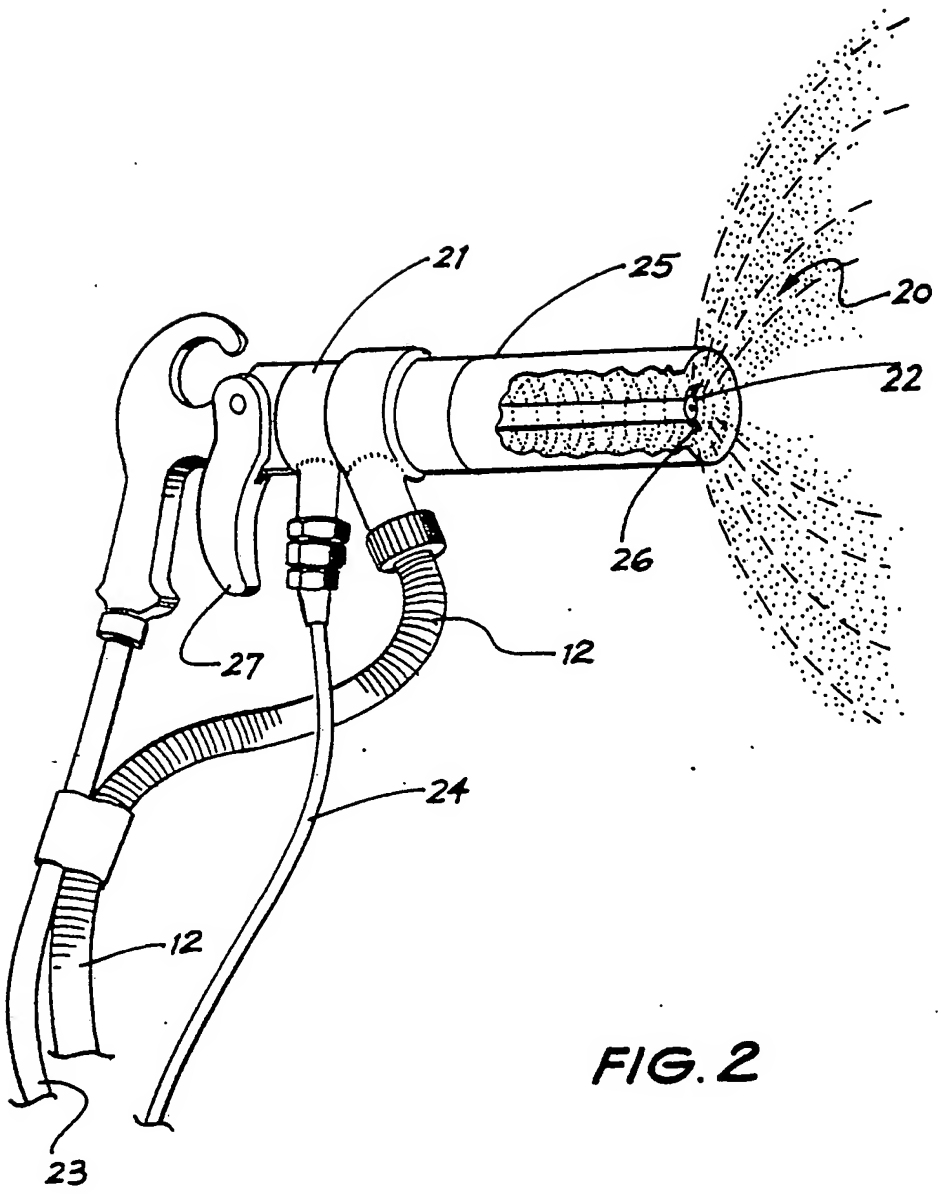

FIG. 2

INTERNATIONAL SEARCH REPORT

International Application No PCT/AU83/00196

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl.[3] B05D 1/34, 1/36, 3/04, 3/10, B05B 5/02, 7/06, 12/08 // B05D 7/00, .7/06, 7/14

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
IPC	B05D 1/34, 1/36, B05B 5/02
US Cl.	427/341, 524/902

Documentation Searched other than Minimum Documentation to the Extent that such Documents are included in the Fields Searched [5]

AU: IPC as above, plus B05D 3/04, 3/10, 7/00, 7/06, 7/14, B05B 7/06, 12/08

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
X	AU, B, 43652/72 (476431) (A.C. HATRICK CHEMICALS PTY. LTD.) 3 January 1974 (03.01.74) (& US, A, 3874898, & DE, A, 2229916, & FR, A, 2143227, & GB, A, 1364359, & JP, A, 48028597)	(1-9)
X	AU, A, 36214/78 (MIDEED PTY. LTD.) 24 November 1979 (24.11.79)	(1,8,13)
P	AU, A, 11964/83 (LIQUID CARBONIC INC.) 8 September 1983 (08.09.83) (& EP, A, 87969, & JP, A, 58163784)	(1,13)
X	EP, A, 0045040 (E.I. DU PONT DE NEMOURS AND COMPANY) 3 February 1982 (03.02.82) (& US 4346144, & JP, A, 57051760)	(1,2,5)
X	US, A, 4105725 (ROSS) 8 August 1978 (08.08.78) (& JP, A, 49095823)	(13)
X	US, A, 3851402 (TURNBULL et al) 3 Decemeber 1974 (03.12.74) (& DE, A, 2357600)	(13)
X	JP, A, 52-145447 (TOYOTA JIDOSHA KOGYO K.K.) 3 December 1977 (03.12.77) (JAPATIC English Language Abstract)	(12)

CONTINUED

* Special categories of cited documents: [15]

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IV. CERTIFICATION

Date of the Actual Completion of the International Search [2]	Date of Mailing of this International Search Report [2]
28 February 1984 (28.02.84)	02 MARCH 1984 (02.03.84)
International Searching Authority [1]	**Signature of Authorized Officer [20]**
Australian Patent Office	A.S. MOORE A.S. Moore

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No [18]
X	DE, A, 1577859 (ERNST MUELLER, LUFT-UND FARBSPRITZTCHNIK) 28 August 1969 (28.08.69)	(12)
A	AU, B, 78531/81 (527297) (ASHLAND OIL, INC.) 24 June 1982 (24.06.82) (& US, A, 4365039)	(12)
X,Y	AU, B, 79217/75 (480111) (RANSBURG CORPORATION) 23 September 1976 (23.09.76) (& GB, A, 1507341, & FR, A, 22665484, & JP, A, 50138038, & US, A, 3930061)	(1,2)